(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization International Bureau



(43) International Publication Date 19 February 2004 (19.02.2004)

(10) International Publication Number WO 2004/014580 A2

(51) International Patent Classification7:

B22C

(21) International Application Number:

PCT/GB2003/003459

(22) International Filing Date: 8 August 2003 (08.08.2003)

(25) Filing Language:

English

(26) Publication Language:

English

(30) Priority Data: 0218382.0

8 August 2002 (08.08.2002)

(71) Applicant (for all designated States except US): THE UNIVERSITY OF BIRMINGHAM [GB/GB]; Research Park, Vincent Drive, Birmingham B15 2SQ (GB).

(72) Inventor; and

(75) Inventor/Applicant (for US only): JONES, Samantha [GB/GB]; 121 Upper Ettingshall Road, Bilston, West Midlands WV14 9QY (GB).

(74) Agent: WARD, David, I.; Marks & Clerk, Alpha Tower, Suffolk Street Queensway, Birmingham B1 1TT (GB).

(81) Designated States (national): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW.

(84) Designated States (regional): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

Published:

without international search report and to be republished upon receipt of that report

for two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

(54) Title: IMPROVED INVESTMENT CASTING PROCESS

(57) Abstract: The present invention provides a method for production of a shell mould. The method comprises the sequential steps of:- (i) dipping a preformed expendable pattern into a slurry of refractory particles and colloidal liquid binder whereby to form a coating layer on said pattern, (ii) depositing particles of refractory material onto said coating, and (iii) drying, steps (i) and (iii) being repeated as often as required to produce a shell mould having a primary coating layer and at least one secondary coating layer, characterised in that during at least one performance of step (ii) a gel-forming material is also deposited onto the coating layer formed in step (i). The invention also relates to a shell mould producible by the method.

- 1 -

IMPROVED INVESTMENT CASTING PROCESS

The present invention relates to an improved investment casting process, and in particular to a process which is much more rapid than conventional processes.

A typical investment casting process involves the production of engineering metal castings using an expendable pattern. The pattern is a complex blend of resin, filler and wax which is injected into a metal die under pressure. Several such patterns, once solidified are assembled into a cluster and mounted onto a wax runner system. The wax assembly is dipped into a refractory slurry consisting of a liquid binder and a refractory powder. After draining, grains of refractory stucco are deposited onto the damp surface to produce the primary refractory coating (the covering of the assembly with refractory material is known as "investing", hence the name for the process). When the primary coat has set (usually by air drying until the binder gels) the assembly is repeatedly dipped into a slurry and then stuccoed until the required thickness of mould shell is built up. Each coat is thoroughly hardened between dippings, and so each mould can take from between 24 and 72 hours to prepare. The purpose of the stucco is to minimise drying stresses in the coatings by presenting a number of distributed stress concentration centres which reduce the magnitude of any local stresses. Each stucco surface also provides a rough surface for keying in the next coating. The particle size of the stucco is increased as more coats are added to maintain maximum mould permeability and to provide bulk to the mould.

In recent years, advanced ceramics (eg. silicon nitride) components have been developed which offer significant advantages over comparable metal components. Many processes by which such ceramic components can be made are known, and these include machining, injection moulding, slip casting, pressure casting and gelcasting. In gelcasting, a concentrated slurry of ceramic powder in a solution of organic monomer is poured into a mould and polymerised in situ to form a green body in the shape of the mould cavity. After demoulding, the green ceramic body is dried, machined if necessary, pyrolysed to remove binder and then sintered to full density. Aqueous based systems, such as the acrylamide system, have been developed in which water-soluble monomers are used, with water as the solvent.

It is an object of the present invention to provide an improved investment casting process which obviates or mitigates one or more problems associated with known investment casting processes and which preferably significantly reduces the time required for forming a shell mould.

According to the present invention, there is provided a process for the production of a shell mould, comprising the sequential steps of:-

- (i) dipping a preformed expendable pattern into a slurry of refractory particles and colloidal liquid binder whereby to form a coating layer on said pattern,
- (ii) depositing particles of refractory material onto said coating, and
- (iii) drying,

steps (i) to (iii) being repeated as often as required to produce a shell mould having the required number of coating layers, characterised in that

during at least one performance of step (ii) particles of a gel-forming material are also deposited onto the coating layer formed in step (i) such that after contact with the coating layer moisture is absorbed by the gel-forming material thereby causing gellation of the colloidal binder so reducing the time required for drying in step (iii).

Preferably, the method also includes the additional step (iv), carried out after the final step (iii) of applying a seal coat comprising a slurry of refractory particles and colloidal liquid binder, followed by drying.

In shell mould formation, the coating layer applied to the expendable pattern is usually referred to as the primary coating and subsequent slurry coatings are referred to as secondary coatings. Typically, three to twelve secondary coatings are applied.

Preferably, the gel-forming material is applied onto each secondary coating (i.e. during each repetition of step (ii) after the first). More preferably, the gel-forming material is applied onto the primary coating.

It will be understood that the deposition of refractory particles and gelforming material in step (ii) may be achieved by any convenient method, such as by use of a rainfall sander or a fluidised bed. The refractory particles and gel-forming material may be applied independently and/or sequentially or preferably they may be premixed. In a particularly preferred embodiment the refractory particles are pre-coated with the gelforming material.

Preferably, the amount of gel-forming material used in step (ii) is no more than 10% by weight, more preferably no more than 5%, even more preferably no more than 3% and most preferably no more than 2wt% of the refractory material particles used in that step (ii).

Preferably, said gel-forming material is a polymer, more preferably a super absorbent polymer exemplified by polyacrylamide and polyacrylate.

In general, at least 50wt% (and even more preferably at least 80wt%) of the gel-forming material particles (in those embodiments in which the gelforming material does not coat the refractory material particles) are preferably no larger than 1mm, more preferably no larger than 300 μ m and most preferably no larger than 200 μ m. In a particularly preferred embodiment, substantially all (i.e. at least 95wt%) of the polymer particles are no more than 300 μ m in size. Although there is no theoretical minimum particle size for the gel-forming material, fine powders can be problematic, particularly when applied by a rainfall sander. Thus, a preferred minimum particle size is 50 μ m and more preferably 75 μ m. The particles may all be substantially the same size, or there may be a particle size distribution below the maximum size.

Advantageously, the process (apart from the use of the moisture absorbing material and the reduced drying times which result) can be substantially the same as a standard investment casting process using conventional machinery and materials. Thus, it will be understood that the nature of the expendable pattern, the slurry compositions used in step (i) (and step (iv)

when present) and the refractory particles used in step (ii) may be any of those known to the person skilled in the art of investment casting.

Moreover, the method preferably includes a step of removing the expendable pattern from the shell mould after the last step (iii) (or step (iv) when present) and more preferably the method includes a final step of firing the resultant shell mould.

Firing may be effected by heating to 950°C or more. Preferably however, a multi-step firing procedure is adopted. For example, a first step may involve heating to a temperature of from 400 to 700°C at a heating rate of from 1 to 5°C/min (preferably 1 to 3°C/min), followed by a second step of heating to at least 950°C (preferably about 1000°C) at a rate of from 5 to 10°C/min. The temperature may be maintained between the first and second steps for a short period (eg. less than 10 minutes). Heating to at least 950°C may be effected in three or more steps.

The present invention further resides in a shell mould producible by the method of the present invention.

The present invention will be further described with reference to the following examples.

Comparative Example 1

The comparative example was intended to be representative of a standard shell used for aluminium alloy casting and was constructed as follows:-

A filled-wax test piece was dipped into a first slurry (primary) for 30 seconds and drained for 60 seconds. Coarse-grained stucco material was then deposited onto the wet slurry surface by the rain fall sand method (deposition height about 2m). The coated test piece was placed on a drying carousel and dried for the required time under controlled conditions of low air movement. Extended drying removes moisture from the colloidal binder, forcing gellation of the particles to form a rigid gel.

Subsequent coats were applied by dipping (30 seconds) in a second (secondary) slurry followed by draining (60 seconds), with subsequent stucco application (rainfall sand method, deposition height about 2m) and drying for the required time after each stucco application. In total, four secondary coatings were applied. Finally, a seal coat was applied (dip in secondary slurry, but no stucco application), followed by drying.

The primary and secondary slurry specifications are contained in Table 1, with the other various process parameters being given in Table 2. The latex addition in Table 1 relates to the use of a water-based latex system, which is added to the base binder to improve unfired strength.

Table 1: Slurry specifications for aluminium shell preparation (all figures are wt %)

	binder silica content (wt%)	latex		refractory
Slurry		polymer addition (wt%)	filler type	loading
				(wt% of total
				slurry)
	26		(a) 200 mesh zircon	77%
Primary	26 6	6	(b) 200 mesh fused	a:b 3:1

			silica	
Seconda ry	22	8	200 mesh fused silica	57%

•

Table 2: Shell build specifications for comparative example

Coating	Stucco	Drying air speed	Drying time
		(ms ⁻¹)	(mins)
primary	50/80 mesh	0.4	1440
	alumino-silicate		
secondary 1	30/80 mesh	3	90
	alumino-silicate		
secondary 2	30/80 mesh	3	90
	alumino-silicate		
secondary 3	30/80 mesh	3	90
	alumino-silicate		
secondary 4	30/80 mesh	3	90
	alumino-silicate		
seal coat	none	3	1440
· · · · · · · · · · · · · · · · · · ·		Total	3240

Example 1

The shell mould according to Example 1 was made in the same manner as for comparative example 1 using the slurries of Table 1, except that the stucco applied onto the secondary coatings included particles of polyacrylamide (at a loading of 1 part polyacrylamide to 10 parts stucco. The process parameters are given in Table 3. When the polyacrylamide is deposited onto the wet slurry surface, it rapidly absorbs moisture from the adjacent colloidal portion of the slurry forcing gellation to a rigid gel without the necessity of extended drying times.

It is anticipated that drying times can be reduced even further by the inclusion of polyacrylamide polymer in the stucco applied to the primary slurry coating.

Table 3: Shell build specifications for Example 1

Coating	Stucco	Drying air	Drying
		speed (ms ⁻¹)	time (mins)
primary	50/80 mesh alumino-silicate	0.4	1240
secondary	30/80 mesh alumino-silicate –	3	10
1	polyacrylamide* (10:1)		
secondary	30/80 mesh alumino-silicate –	3	10
2	polyacrylamide* (10:1)		
secondary	30/80 mesh alumino-silicate –	3	10
3	polyacrylamide* (10:1)		
secondary	30/80 mesh alumino-silicate –	3	10
4	polyacrylamide* (10:1)		
seal coat	none	3	10
		Total	1490

^{*} particle size 86wt% > 1mm, 500μm ≤ 14wt% ≤ 1mm

The shell mould of Example 1 is less dense and uniform in comparison with comparative example 1. The shell of Example 1 is more open and delaminated in places due to swelling of the individual polymer particles during absorbance of moisture from the colloidal binder. The large particle size is disadvantageous in this respect and it is anticipated that these defects will be much reduced by the use of a smaller and much more controlled particle size polyacrylamide addition to the standard stucco sizes.

Shell Thickness Comparisons

Comparisons of the ceramic shell thickness achieved for acrylamide modified (Example 1) and standard (comparative example 1) shell systems can be seen in Table 4. The polyacrylamide increases the shell thickness because the particle size is much larger than the stucco itself. The large

size is also represented by the relatively large standard deviation in the data.

Table 4: shell thickness comparison

	-4-4	No. of	Average Thickness	standard
	status	samples	(mm)	deviation (mm)
Example 1	unfired	5	6.81	0.92
Comparative Example 1	unfired	10	4.60	0.26

Room Temperature Flat Bar Strength Measurement

Strength measurements were carried out in accordance with BS 1902. Injected wax bars were used as the formers for the ceramic shells formed by the procedures indicated above. After formation, the shells were steam Boilerclave (TM) de-waxed at 8 bar pressure for 4 minutes, followed by a controlled de-pressurisation cycle at 1 bar/minute. Test pieces, approximately 20mm x 80mm were cut using a grinding wheel and tested in a 3 point bend mode at room temperature (primary coat in compression).

A comparison of the maximum strengths achieved at room temperature in the 3-point bend mode for the shell samples is shown in Table 5. The high dry, green strength of the comparative example 1 shell is a direct result of the latex polymer content, which is reflected by the reduction in strength as the sample is fired at 1000°C and the latex burns out (data not shown). The strength of the Example 1 shell is relatively low, which is a direct result of the delamination and defects introduced by the use of a very large particle size polyacrylamide. It is anticipated that by the use of a smaller polymer particle size, the swelling of the acrylamide polymer

should be reduced to a level which would be more acceptable for investment casting.

Table 5: flat bar fracture strength

Sample	test piece	status	fracture strength
Comparative Example 1	flat bar	green, dry	7.8 +/-0.7
Example 1	flat bar	green, dry	2.2 +/-0.9

Example 2

In order to address the above-mentioned problems, a further example was prepared, the key differences with Example 1 being:-

- (i) a smaller particle size of more absorbent polymer was employed,
- (ii) a smaller amount of polymer was used, and
- (ii) polymer was incorporated into the primary stucco coating.

The shell build specifications are given in Table 6 below. The slurries were as shown in Table 1.

Table 6: Shell build specifications for Example 2

Coating	Stucco	Drying air	Drying
		speed (ms ⁻¹)	time (mins)
primary	50/80 mesh alumino-silicate	1.8	10
	Liquiblock 144 (2.5wt%)*		
secondary	30/80 mesh alumino-silicate	3	10
1	Liquiblock 144 (2.5wt%)*		
secondary	30/80 mesh alumino-silicate	3	10
2	Liquiblock 144 (2.5wt%)*		
secondary	30/80 mesh alumino-silicate	3	10
3	Liquiblock 144 (2.5wt%)*		
secondary	30/80 mesh alumino-silicate	3	10
4	Liquiblock 144 (2.5wt%)*		

seal coat	none	3	10
		Total	60

 $^{^{*}}$ polyacrylamide having particle size $< 300 \mu m$

The green dry strength for Example 2 was measured as 2.83 +/-0.63 MPa. This was obtained using a different rain sand system than for Example 1, the sand being deposited from a lower height (approximately 10 cm) which is known to reduce strength values. For comparison, comparative example 1 was repeated (referred to hereinafter as comparative example 2) and found to have a green dry strength of 4.86+/-0.54 MPa. Thus, it has been found that in less than 2% of the time required to produce a standard shell mould, the method of the present invention allows the production of a mould having nearly 60% of the strength, which is, as will be shown below, sufficient for casting.

In addition to the green dry strength measurements, Example 2 and comparative example 2 were tested for their green wet strength (to simulate strength during de-waxing) and their fired strength under different heating regimes. The results are shown in Table 7 below.

Table 7: flat bar fracture strengths for Example 2

Example	Status	Fracture Strength (MPa)
	green, dry	4.86+/-0.54
C C C C C C C C C C C C C C C C C C C	green, wet	4.55 + /-0.47
Comparative Example 2	Fired (method A)	4.24+/-0.61
	Fired (method B)	3.80+/-0.38
Example 2	green, dry	2.83 + /-0.63
	green, wet	2.47 + /-0.43

Fired (method B)	2.17+/-0.13
Fired (method C)	2.03 +/-0.45

Firing method A: to 1000°C @20C/min, dwell 60 min, furnace cool Firing method B: to 700°C @ 1C/min, dwell 6 min, to 1000°C @5C/min, dwell 30 min, furnace cool

Firing method C: to 700°C @ 2C/min, dwell 6 min, to 1000°C @10C/min, dwell 60 min, furnace cool.

The Example 2 moulds did not crack during de-waxing. Thus, it has been shown that the method of the present invention allows the production of shell moulds, which are sufficiently strong for investment casting, in a fraction of the time required using standard methods.

- 14 -**CLAIMS** 1. A method for the production of a shell mould, comprising the sequential steps of:-(i) dipping a preformed expendable pattern into a slurry of refractory particles and colloidal liquid binder whereby to form a coating layer on said pattern, depositing particles of refractory material onto said coating, (ii)

- and
- (iii) drying, steps (i) to (iii) being repeated as often as required to produce a shell mould having a primary coating layer and at least one secondary coating layer, characterised in that during at least one performance of step (ii) a gel-forming material is also deposited onto the coating layer formed in step (i).
- The method as claimed in claim 1, wherein the method also 2. includes the additional step (iv), carried out after the final step (iii), of applying a seal coat comprising a slurry of refractory particles and liquid binder, followed by drying.
- The method as claimed in claim 1 or 2, wherein the gel-forming 3. material is applied onto each secondary coating.
- The method as claimed in any preceding claim, wherein the gel-4. forming material is applied onto the primary coating layer.

- 6. The method is claimed in claim 5, wherein the polymer is polyacrylamide or polyacrylate.
- 7. The method as claimed in claim 5 or 6, wherein the polymer is a particulate material and at least 50wt% of the polymer particles are 300µm or smaller.
- 8. The method as claimed in claim 7, wherein at least 95wt% of the polymer particles are 300μm or smaller.
- 9. The method as claimed in any one of claims 1 to 5 wherein the refractory particles are coated with gel-forming material.
- 10. The method is claimed in any preceding claim which includes a step of removing the expendable pattern from the shell mould after the last step (iii) or step (iv) when present and preferably a final step of firing the resultant shell mould.
- 11. The method as claimed in claim 10, wherein firing is effected by heating to a temperature of from 400 to 700°C of a heating rate of from 1 to 5°C/min, followed by heating to at least 950°C at a heating rate of 5°C/min or more.

layers comprises a gel-forming material, refractory particles and gelled

colloidal liquid binder.

- The shell mould as claimed in claim 15, wherein the gel-forming 16. material is a super absorbent polymer.
- The shell mould as claimed in claim 16, wherein said polymer is 17. polyacrylamide.
- The shell mould is claimed in claim 16 or 17, wherein at least 18. 95wt% of the polymer particles are 300µm or smaller.
- The shell mould as claimed in anyone of claims 15 to 18, wherein 19. the amount of gel-forming material in any layer is no more than 10% by weight of the refractory particles in that layer.

ABSTRACT

IMPROVED INVESTMENT CASTING PROCESS

The present invention provides a method for the production of a shell mould. The method comprises the sequential steps of:-

- (i) dipping a preformed expendable pattern into a slurry of refractory particles and colloidal liquid binder whereby to form a coating layer on said pattern,
- (ii) depositing particles of refractory material onto said coating, and
- (iii) drying, steps (i) to (iii) being repeated as often as required to produce a shell mould having a primary coating layer and at least one secondary coating layer, characterised in that during at least one performance of step (ii) a gel-forming material is also deposited onto the coating layer formed in step (i).

The invention also relates to a shell mould producible by the method.